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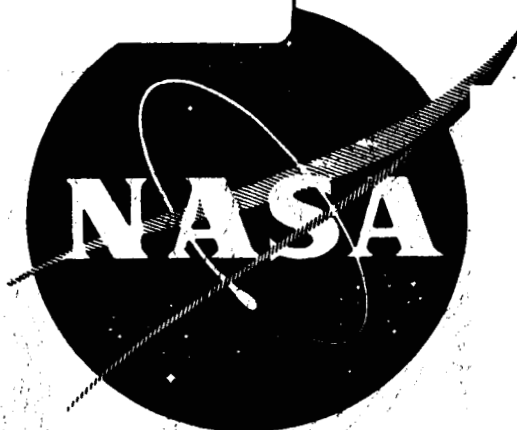
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STUDIES OF ALKALI METAL CORROSION ON MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

Quarterly Progress Report No. 3
For Quarter Ending March 26, 1965

By
R.W. HARRISON

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-6012

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL  ELECTRIC
CINCINNATI, OHIO 45215

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STUDIES OF ALKALI METAL CORROSION ON
MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

QUARTERLY PROGRESS REPORT 3

Covering the Period
December 26, 1964 to March 26, 1965

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I. INTRODUCTION

The program reviewed in this third quarter progress report, covering from December 26, 1964 to March 26, 1965, is sponsored by the National Aeronautics and Space Administration. Its purpose is to examine the influence of stress on the corrosion behavior of an advanced refractory alloy in potassium (Task I) and to investigate corrosion mass transfer effects in a stainless steel-columbium alloy-potassium system (Task II).

Task I

While there is considerable evidence that refractory alloys have excellent corrosion resistance to potassium, there are few experiments which describe the possible effects of stress on corrosion when the stress is sufficiently large to produce substantial amounts of creep during the test. It is appropriate for comparative purposes to study an advanced refractory alloy which has demonstrated excellent corrosion resistance to refluxing potassium during long-time exposures conducted at relatively low stresses at 2000°F. In this regard, D-43 columbium base alloy, in the form of welded capsules, has been tested in potassium under refluxing conditions for periods of 5,000 and 10,000 hours at temperatures on the order of 2000°F (Ref. 1) and has been selected for inclusion in this program.

The D-43 alloy reflux capsules will be tested under conditions which result in about 5 to 10% strain during a 500- to 2,000-hour exposure period in the 2000° to 2200°F temperature range. The reflux capsules used in this study will be of similar size to those previously described (Ref. 1). The capsule wall is reduced in the potassium liquid region and in the vapor condensing region to provide gauge sections where the extent of creep can be measured. Moderate temperature adjustments can be made during the test to achieve the desired strain-time conditions.

Task II

The use of stainless steel, rather than refractory alloys, for power plant radiator construction and for the lower temperature portion of experimental facilities constitutes material and fabrication cost savings. Two methods of employing this approach are: use of co-extruded, stainless steel shell-refractory alloy

core, tubing in the radiator or use of an all stainless steel radiator joined to the system by a bimetallic joint. Although the latter approach is preferred considering cost and problems associated with fabrication and joining of co-extruded tubing, a major uncertainty and limitation arises from the mass transfer of interstitial elements from the stainless steel to the refractory alloys through the alkali metal.

It is well established that the carbon and nitrogen transfer from Type 316SS to Cb-1Zr alloy at temperatures near 1500°F (Ref. 2). While some important aspects of this mass transfer behavior have been examined, several critical details require additional investigation. There is a need to define acceptable time and temperature conditions of operation in terms of maintaining satisfactory performance of the refractory alloys, such as Cb-1Zr alloy. Also, there are certain metallurgical aspects of this behavior which should be investigated in an effort to eliminate or reduce the mass transfer rate. In the latter category, it is most appropriate to consider the stabilization of carbon and nitrogen in the stainless steel by the addition of metallic elements which form carbides and nitrides of high thermodynamic stability. Commercially available, titanium stabilized, Type 321SS is one such alloy. A comparative investigation of this alloy and Type 316SS should indicate the ability of the titanium addition to reduce or eliminate interstitial mass transfer in a stainless steel-Cb-1Zr alloy bimetallic system. Columbium-1% zirconium alloy specimens have been exposed to liquid potassium in Type 321SS and Type 316SS capsules for 1,000 hours at 1400°F under isothermal conditions to evaluate this premise.

II. SUMMARY

During the third quarter of this program, the topics abstracted below were covered. The results are interpretatively presented in this report.

Task I - Stress Corrosion Reflux Capsule Tests

A D-43 alloy reflux stress-corrosion capsule was fabricated, filled with purified potassium, sealed and installed in the test facility.

Assembly and instrumentation of the Task I test facility was completed and the system was evacuated to a pressure of 2×10^{-8} torr prior to the bakeout cycle. The initiation of the test is pending receipt of tungsten caps which are to be installed over the Al_2O_3 probes to prevent any possible reaction between the Al_2O_3 and the D-43 alloy.

Task II - Bimetallic Isothermal Capsule Tests

Two Type 321SS and two Type 316SS capsules, each containing two Cb-1Zr alloy test specimens, were tested isothermally at 1400°F for 1,000 hours without difficulty. Post-test evaluation of capsules has shown that Type 321SS has a significant advantage over Type 316SS in refractory metal-stainless steel-potassium systems in inhibiting mass transfer of the interstitial elements carbon and nitrogen from the stainless steel to the refractory metal.

III. TASK I - STRESS CORROSION REFLUX CAPSULE TESTS

A. Capsule Fabrication and Filling

Approval was received from the NASA Technical Manager to incorporate a boiling nucleator in the D-43 alloy reflux capsule design. Subsequently, components for the nucleators were machined for each of the three D-43 alloy capsules; one complete capsule has been fabricated, Figure 1, leak checked, the welds radiographed and the capsule filled with potassium. The potassium was transferred directly to the capsule from the final hot trapping container and the capsule sealed by electron beam welding in a vacuum of 7×10^{-6} torr. The potassium that was used for filling the capsule was sampled at the same time that the capsule was filled and analyzed for oxygen by the mercury amalgamation method; the results showed the oxygen in the potassium taken from the fill tube to be 5 ppm and the oxygen in the potassium taken from the chamber samples to be 8 ppm. The filled and sealed capsule was examined radiographically to assure a sound electron beam weld and subsequently was installed in the test facility.

B. Test Facility

The high-vacuum LVDT's from Automatic Timing and Controls, Inc. and the high-purity alumina probes from American Lava Company were received during the report interim. In order to help minimize the pump-down time of the Task I facility as a result of outgassing, the LVDT's were heated to 650°F in a high pumping capacity vacuum furnace; after three hours at temperature, a vacuum of 1×10^{-5} torr was achieved. The assembly of all the components in the high-vacuum chamber was completed on 2-24-65 and the system was closed and evacuated. Mass spectrometer leak checking indicated no leaks present and a pre-bakeout vacuum of 2×10^{-8} torr was attained.

At the request of the NASA Technical Manager, the initiation of the test has been delayed pending installation of tungsten caps over the Al_2O_3 probes. The tungsten caps will be utilized as a precautionary measure to avoid the possibility of any deleterious reaction between the Al_2O_3 probes and the D-43 alloy capsule wall. The caps have been designed in order to be compatible with the existing Al_2O_3 probe configuration and the order placed. Delivery is expected on 4-2-65.

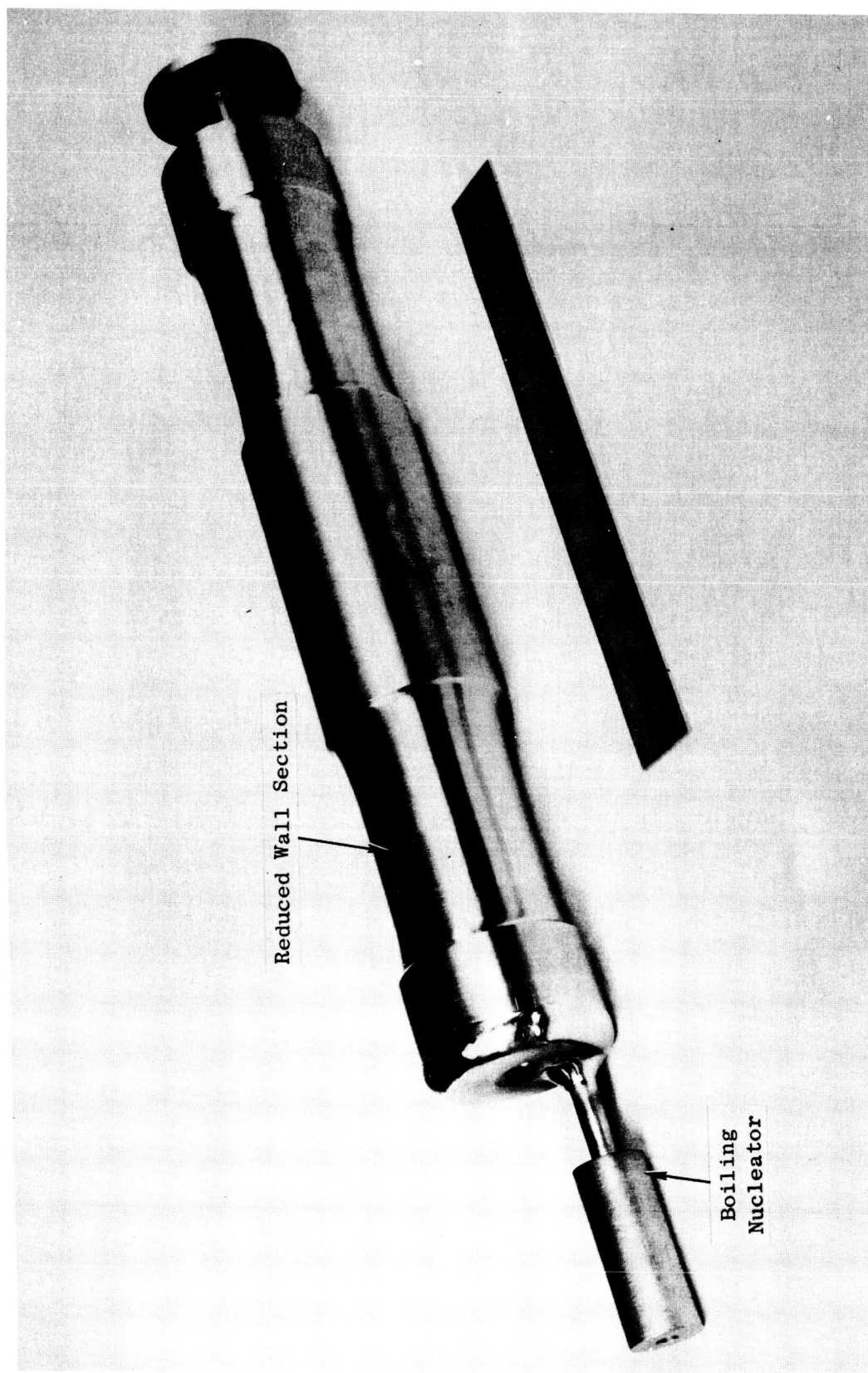


Figure 1. D-43 Alloy Reflux Corrosion Capsule Ready for Filling
C65020211
with Potassium.

The assembly of the strain measuring LVDT-probe units without the tungsten cap is depicted in Figures 2, 3 and 4. The stainless steel spring is utilized to maintain a negligible load contact of the probe on the capsule wall. The square hole in the stainless steel case and the square shank on the probe serve a similar purpose by preventing loss of contact by rotation of the probe. These assembled units are shown in place in the test facility in Figure 5. Adjustments in all planes are provided such that the probes can be located directly on the 0.020-inch thick reduced wall section of the capsule.

The capsule is supported in a Cb-1Zr alloy cup insulated from the support table by tantalum shielding; heating is achieved by use of a split tantalum element. The probes extend through the gap in this element to contact the capsule in the liquid zone, Figure 5. Final location of the probes on the 0.020-inch constant wall thickness in the liquid and condensing zones was achieved by use of a dial gauge, subsequent to adjustment of the capsules in the upper capsule support to maintain capsule verticality, Figure 6. The boiling nucleator is heated by means of a tantalum-sheathed resistance element which is shielded with tantalum sheet, Figure 7. The completed assembly for one capsule with tantalum shielding in place is shown in Figure 8. Using a similar configuration, it is possible to test two capsules simultaneously. Although not shown, the W-Re thermocouples were attached to a vacuum feedthrough, Figure 9, composed of W-25%Re and W-3%Re wires. A demodulator-recorder circuit, in conjunction with secondary LVDT bucking coils to be used as nulling devices, Figure 10, has been instrumented so that the strain can be recorded continuously. A deflection in the capsule wall as small as 0.0001 inch can be measured.

The first capsule test will be initiated under the conditions which were determined from the creep evaluation of the D-43 alloy bar procured for this program. The initial temperature has been established at 2200°F with subsequent adjustments, based upon the actual creep measurements made on the reduced section of the capsule wall, to achieve approximately 5% strain in 1,000 hours.

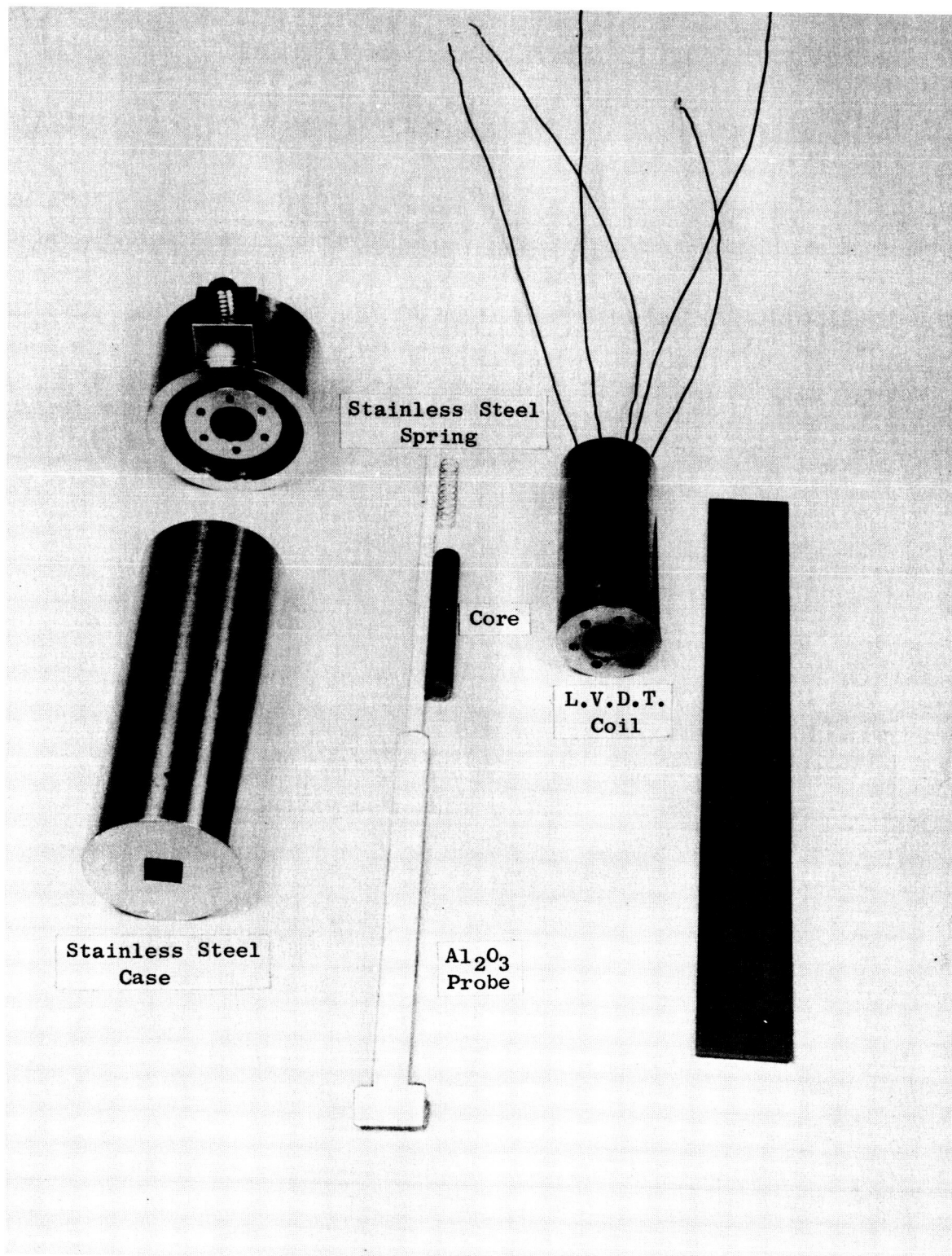


Figure 2. Disassembled Strain Measuring LVDT-Probe Unit. C65020208

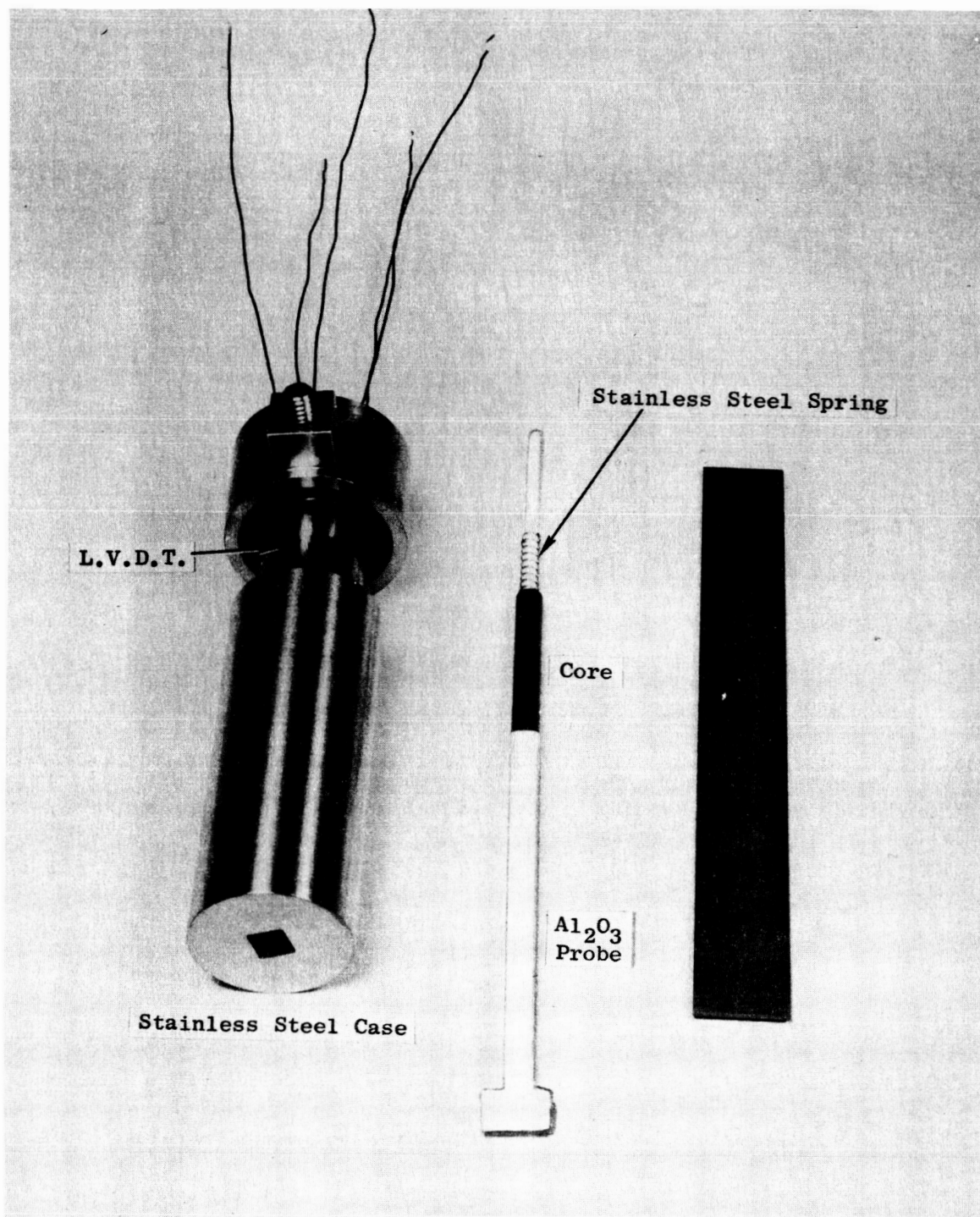


Figure 3. Partially Assembled LVDT-Probe Unit. C65020206

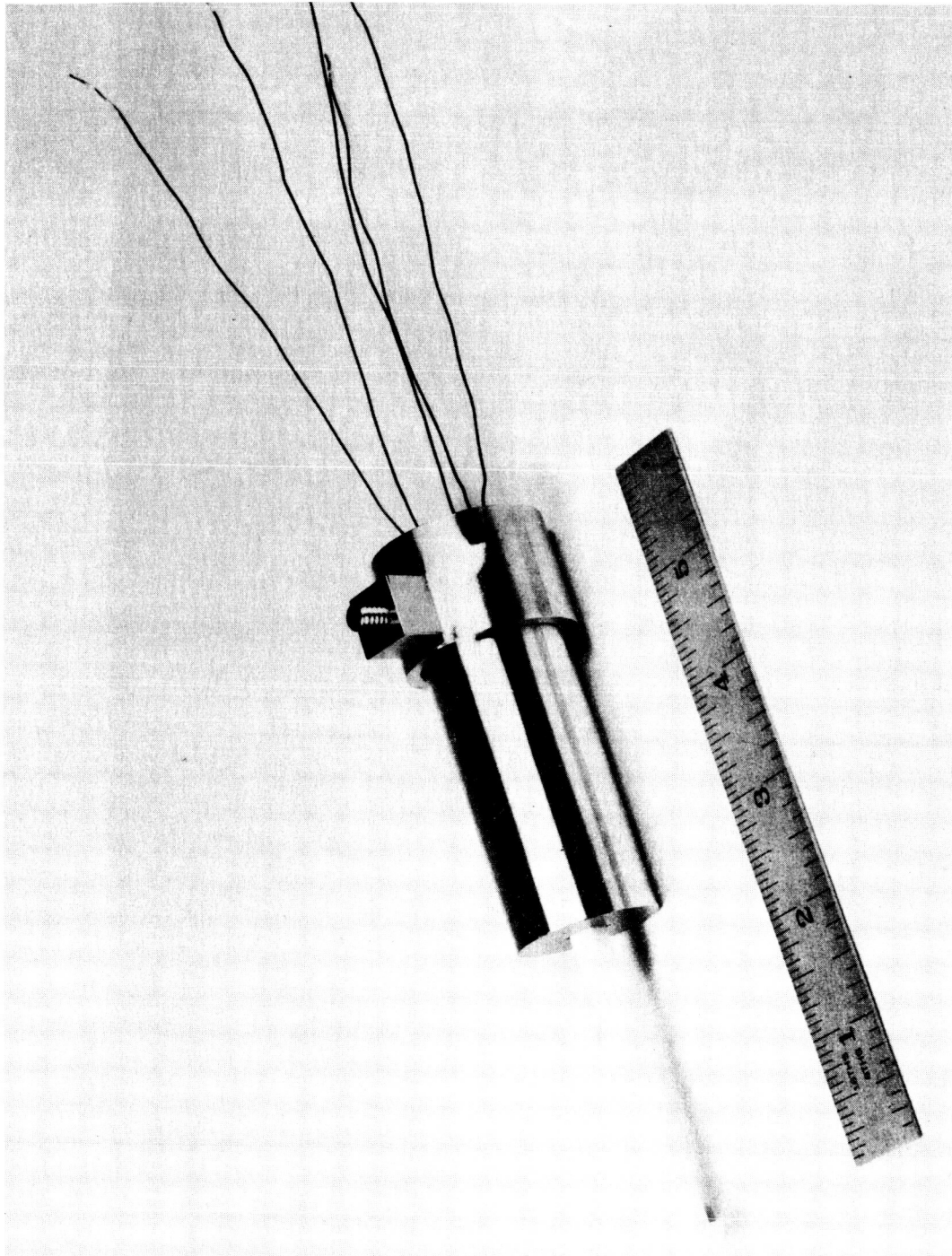


Figure 4. Assembled LVDT-Probe Unit for Strain Measurement. C65020210

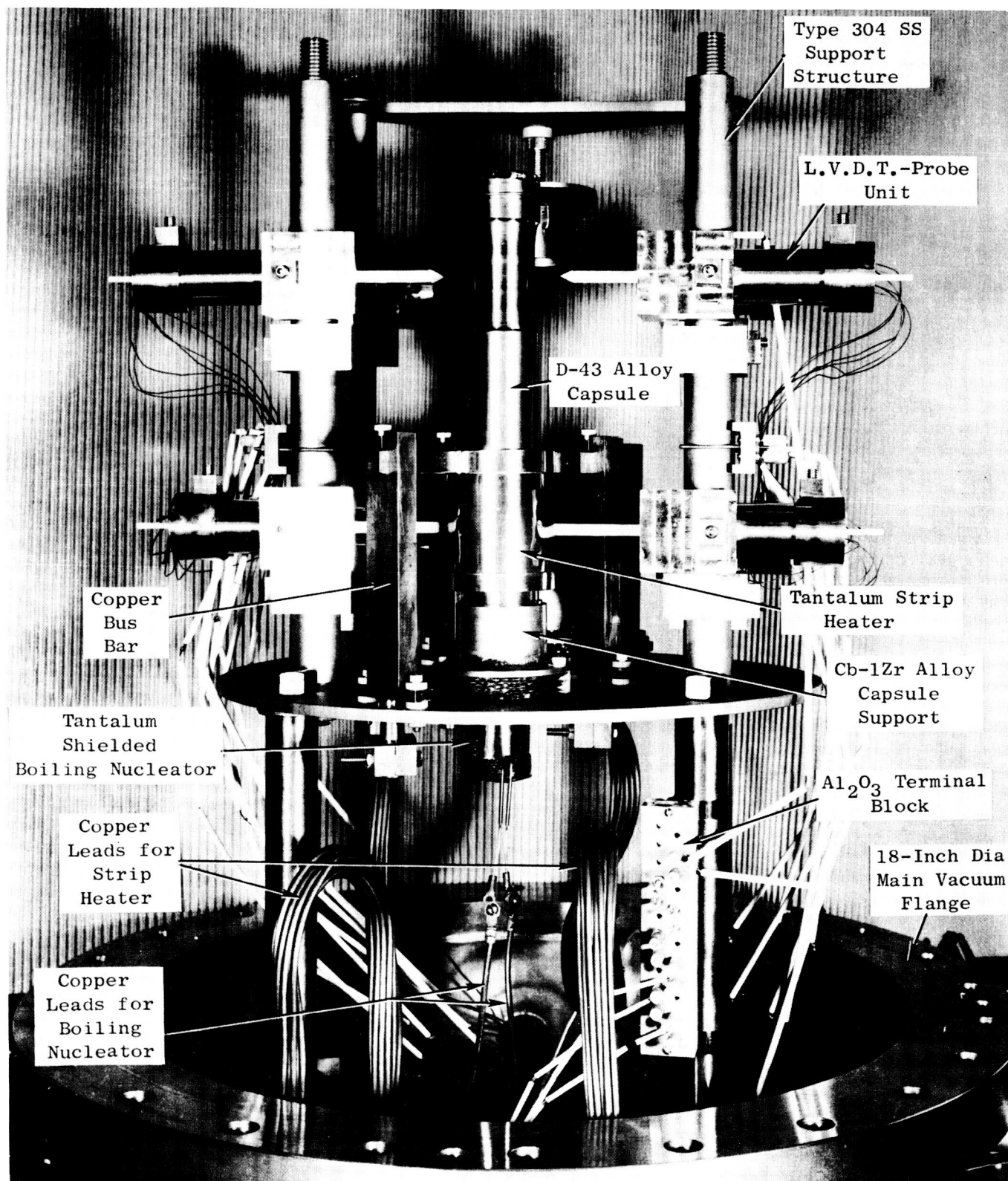


Figure 5. LVDT-Probe Units Installed with a D-43 Alloy Reflux Corrosion Capsule and Tantalum Strip Heating Element in Place in the High-Vacuum Chamber. Strain is Measured in the Condensing Zone and in the Liquid Zone. C65022603

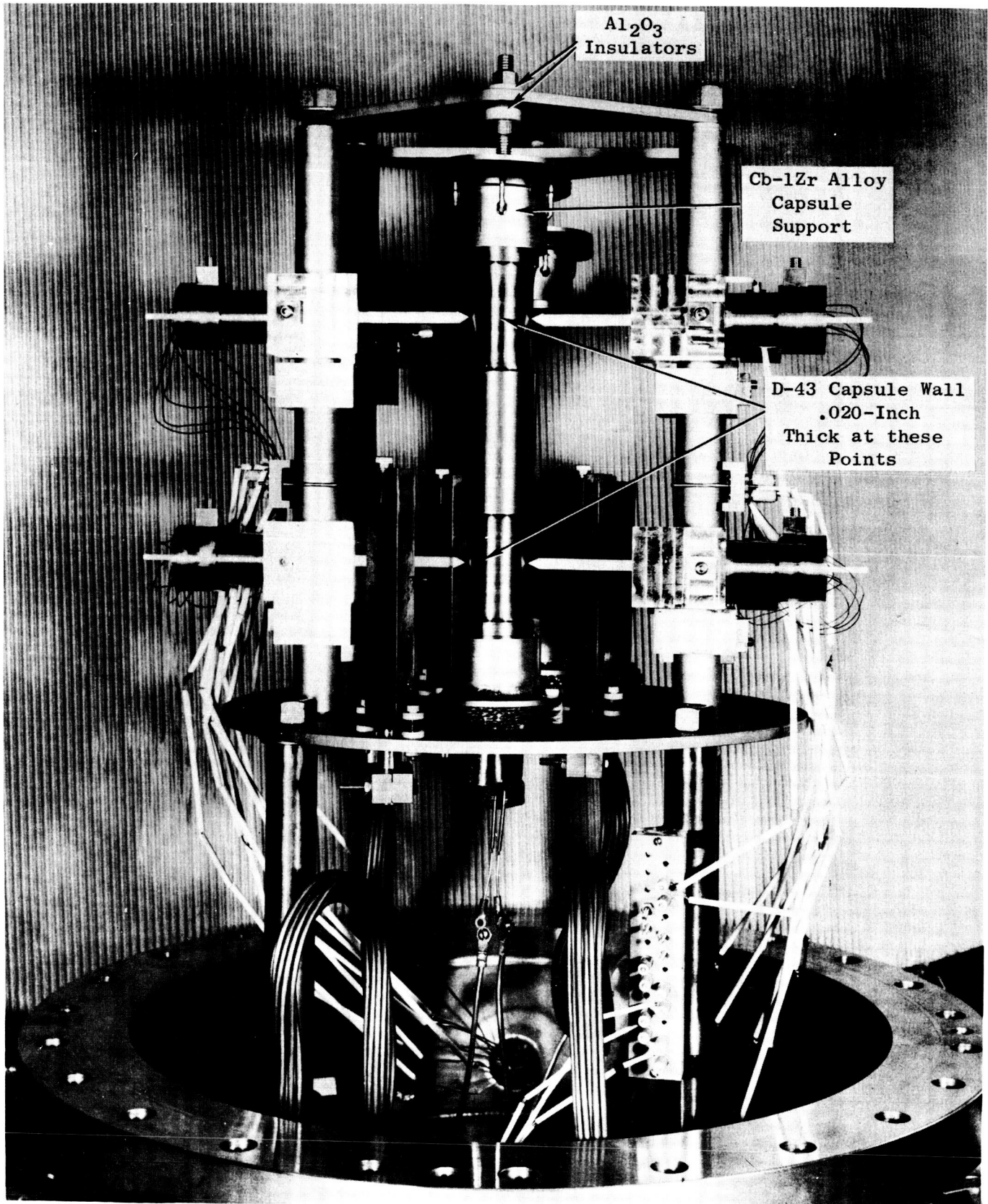


Figure 6. LVDT-Probes Located on Reduced Wall Section. Constant 0.020-Inch Thick Wall Section Located by Means of a Dial Gauge. Upper Cb-1Zr Alloy Capsule Support Maintains Verticality of Capsule and Lateral Position. C65022604

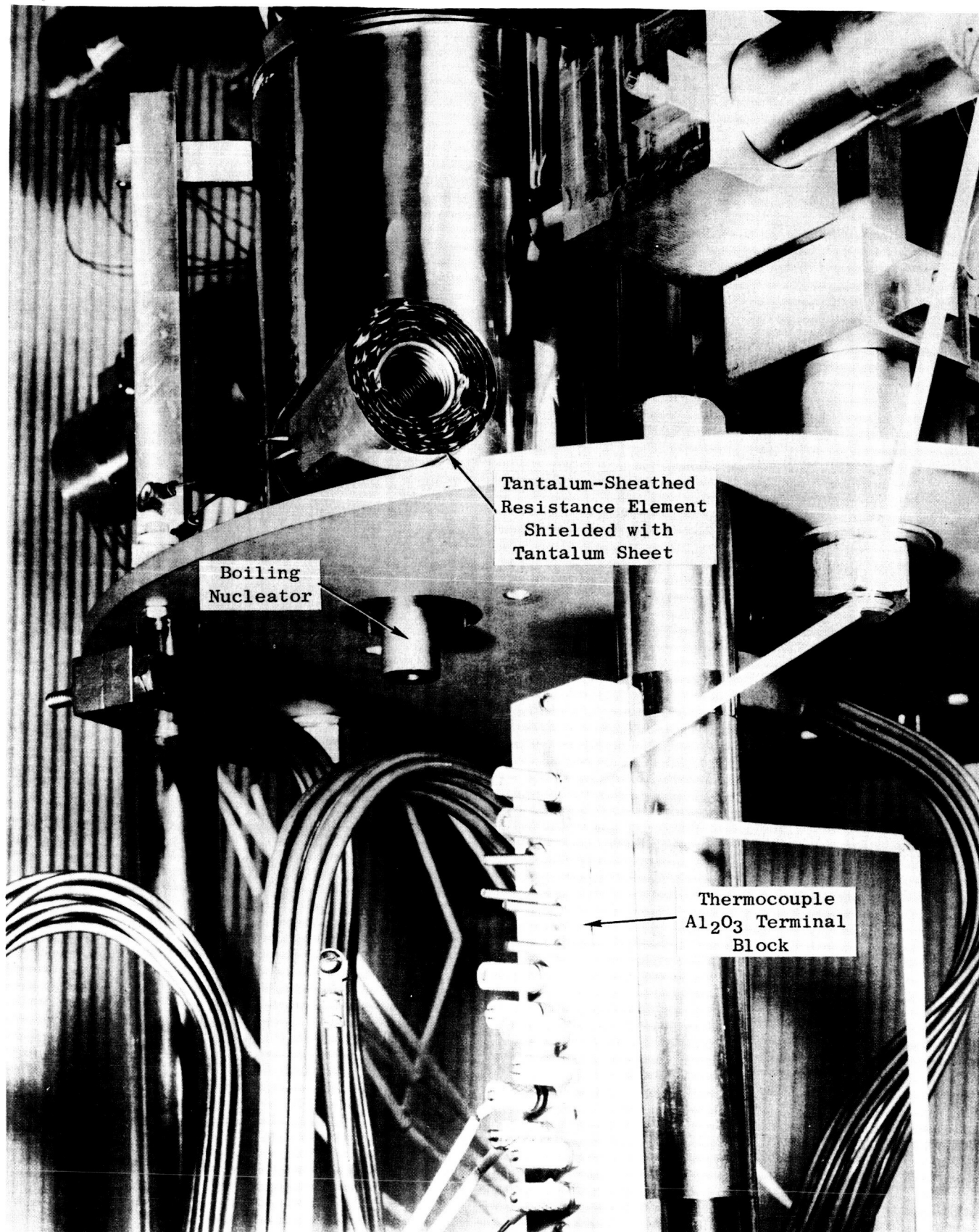


Figure 7. View of Bottom of D-43 Alloy Reflux Corrosion Capsule Extending Through Support Structure. The Boiling Nucleator is Heated by a Tantalum Sheathed Resistance Element.

C65022608

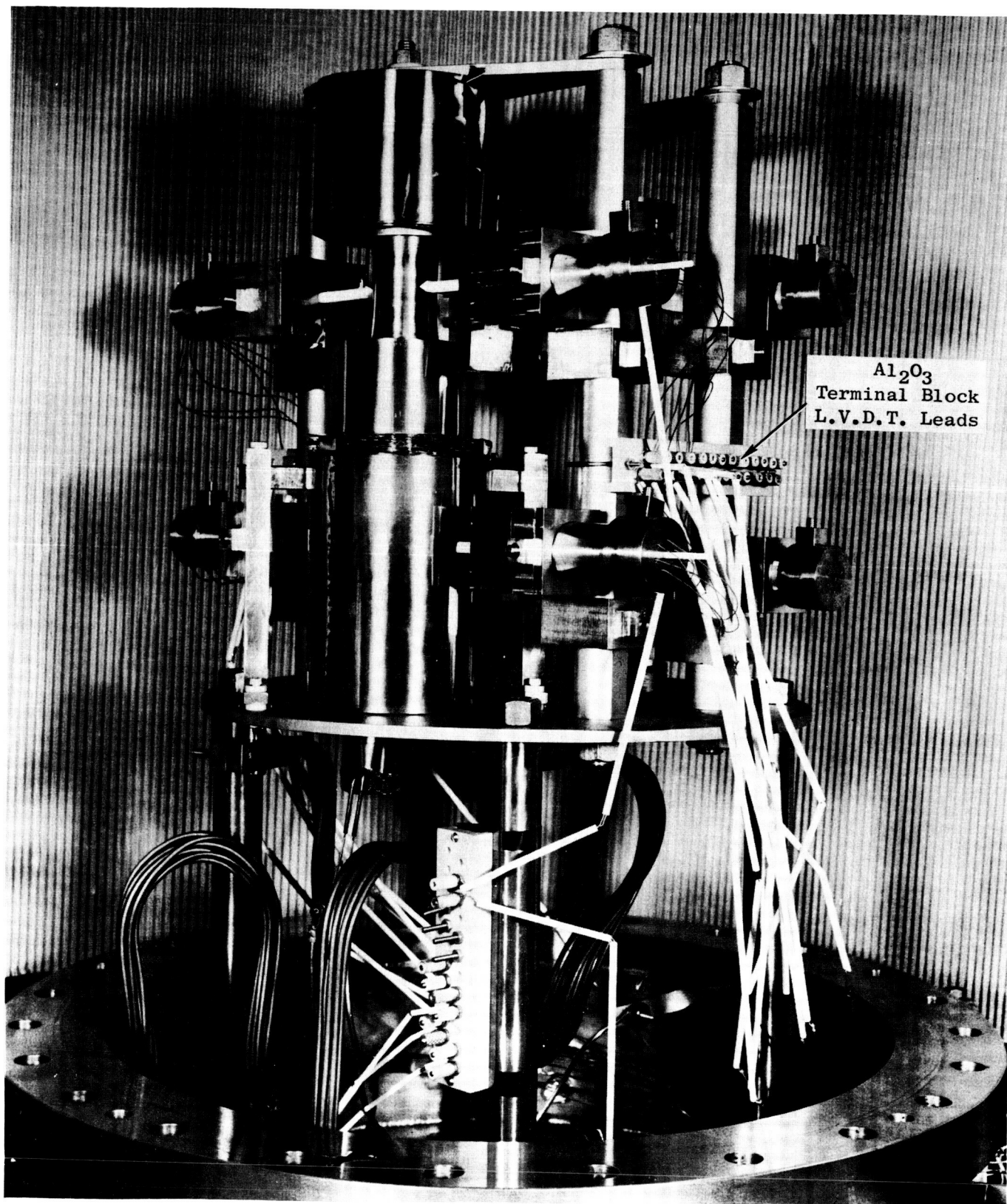


Figure 8. Completed D-43 Alloy Reflux Corrosion Capsule Assembly Showing all Tantalum Heat Shields in Place. Provision for Testing Two Capsules is Provided as Shown. C65022606

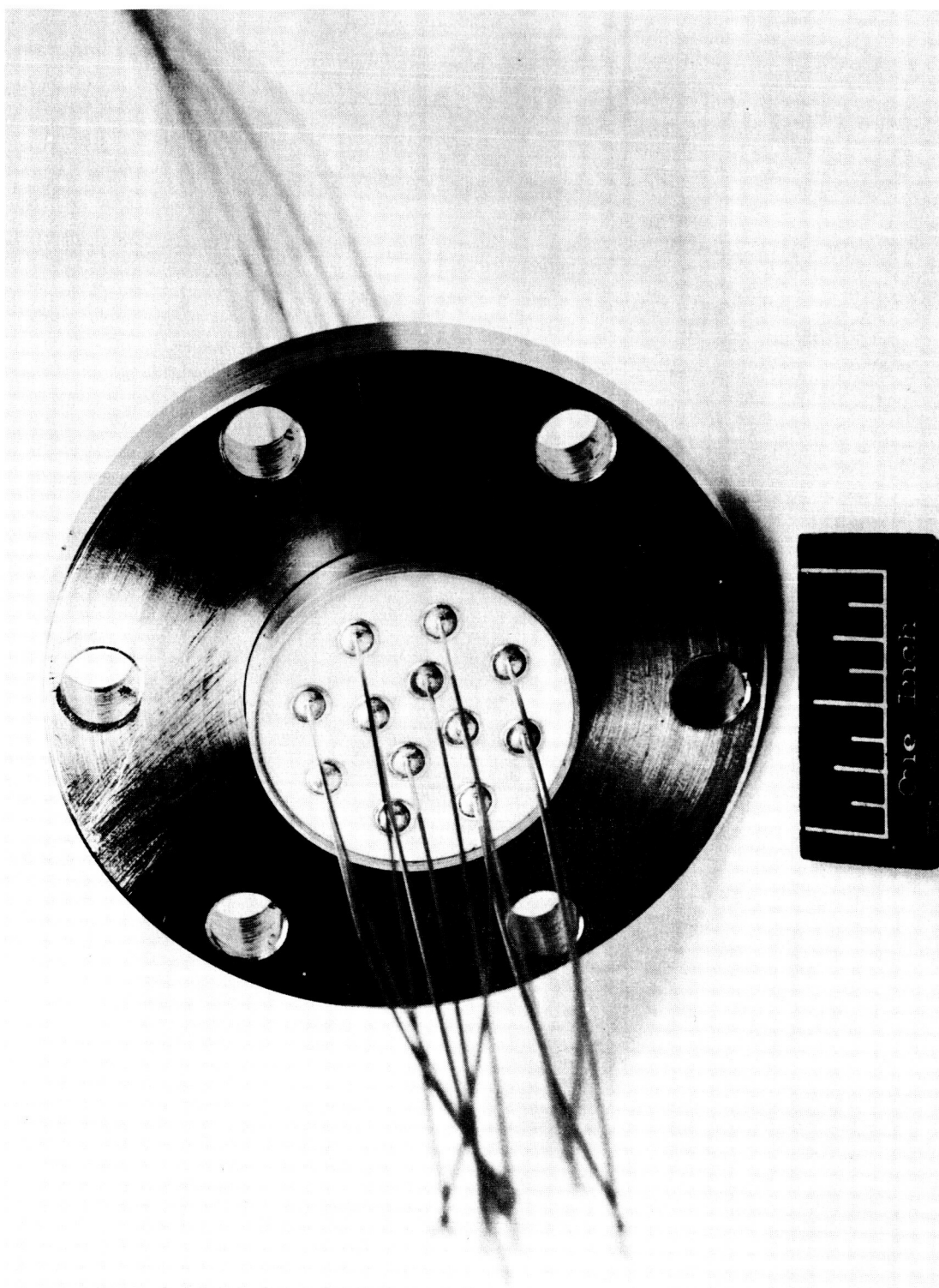


Figure 9. W-Re Feedthrough with Provision for Six W-25%Re vs W-3%Re Thermocouples.
C64111836

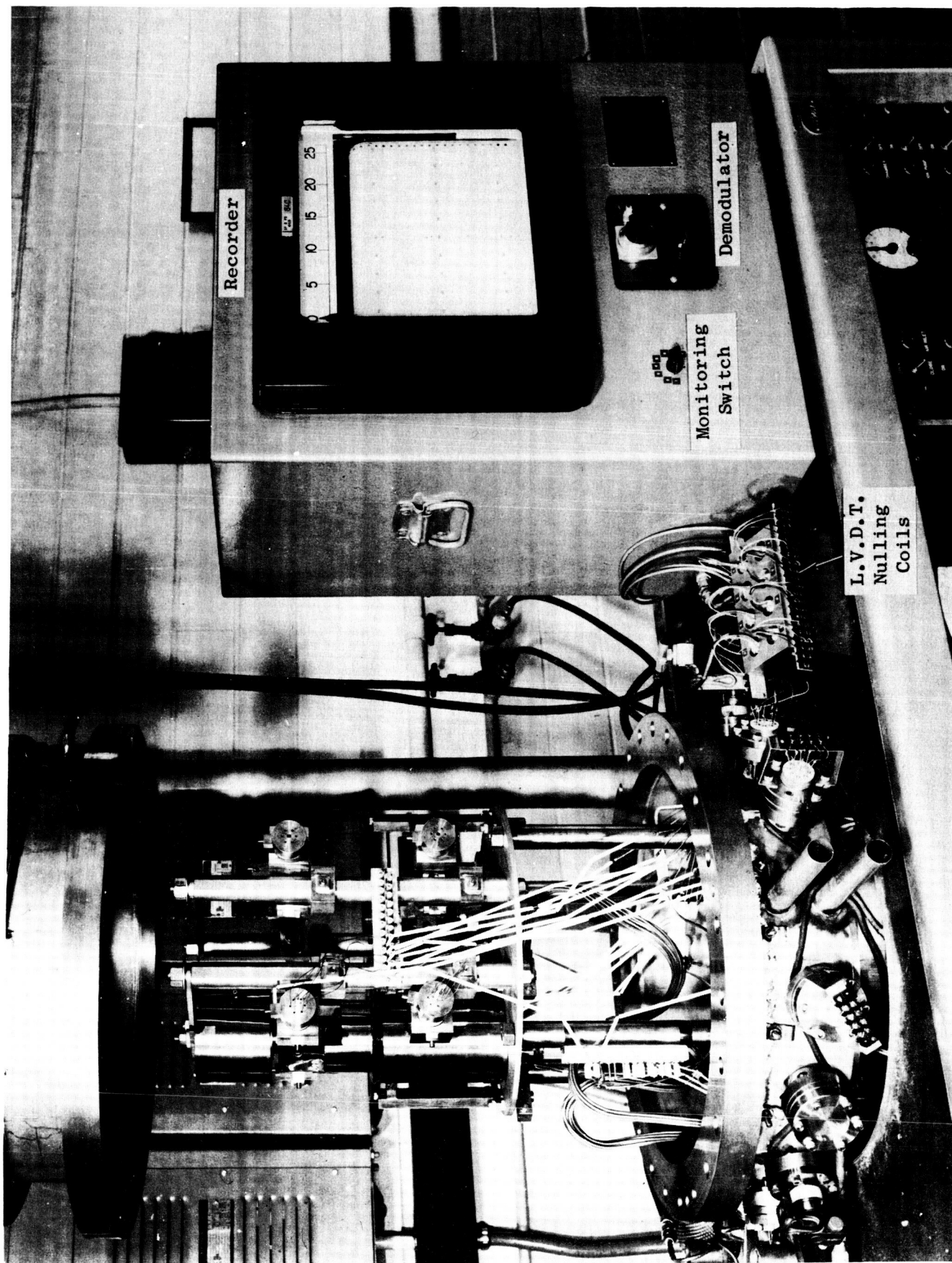


Figure 10. Test Facility to Measure Biaxially Induced Creep in
D-43 Alloy Reflux Corrosion Capsules. C65022609

IV. TASK II - BIMETALLIC CAPSULE PROGRAM

A. Capsule Testing

Two Type 321SS and two Type 316SS capsules, each containing one stress-rupture and one tensile specimen of Cb-1Zr alloy sheet in potassium, were exposed for 1,000 hours at 1400°F in an air environment. A maximum temperature variance of 20°F from the mean temperature of 1405°F was measured for this time period. The mean temperature was calculated on the basis of temperature readings from seven beaded, chromel-alumel thermocouples spot welded to the sides of the capsules. A temperature differential of 20°F also was measured between the top and bottom of one capsule. The capsules were removed from the test facility and examined. As anticipated no leaking was evident and oxidation was minor, Figure 11.

B. Test Evaluation

Post-test evaluation tests that have been completed on the stainless steel capsules and Cb-1Zr alloy test specimens have definitely indicated the advantages of Type 321SS over Type 316SS, with respect to mass transfer of the interstitial elements, for use in refractory alloy-stainless steel-potassium systems.

The stainless steel capsules were opened in an argon atmosphere and drained of potassium. After removal of the Cb-1Zr alloy sheet specimens, the capsules and specimens were cleaned and examined. No appreciable differences in appearance of the internal surfaces of any of the capsules were noted from visual observation, Figure 12. However, a definite discoloration was noted on the Cb-1Zr alloy specimens which were exposed in the Type 316SS, Figure 13, capsules. This discoloration was not evident on the Cb-1Zr alloy specimens exposed in the Type 321SS capsules. Also, a 300 ppm weight increase was measured for a specimen exposed in a Type 316SS capsule; no increase could be detected in a similar specimen exposed in a Type 321SS capsule.

Subsequently, the Cb-1Zr alloy specimens were analyzed for carbon, oxygen, nitrogen and hydrogen and the data are presented in Table I. The chemical analyses further clarify the preliminary observation. Negligible changes in nitrogen and carbon

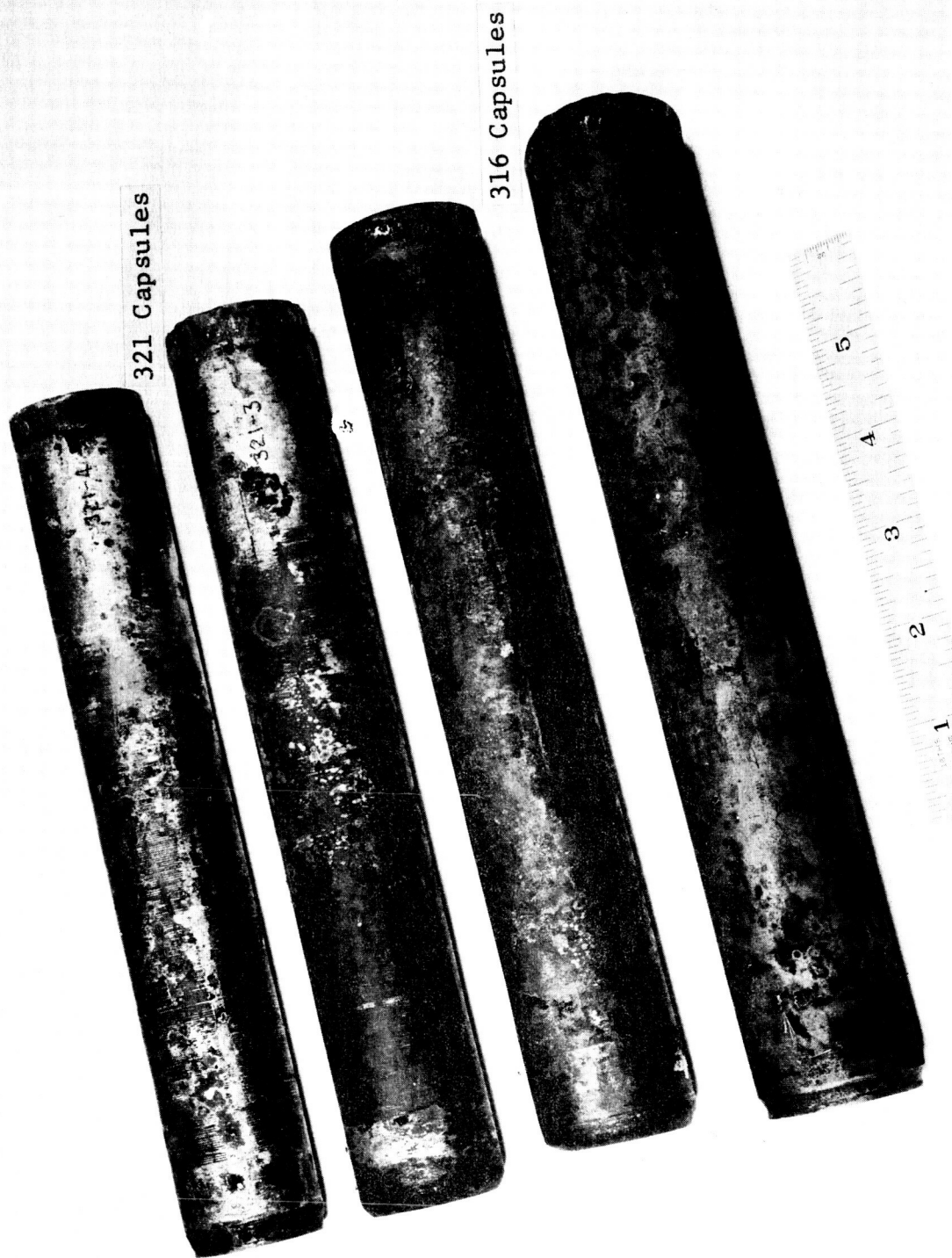


Figure 11. Type 316SS and Type 321SS Isothermal Corrosion Capsules, Containing Potassium and Cb-1Zr Alloy Test Specimens, After Being Exposed for 1,000 Hours at 1400°F in Air.

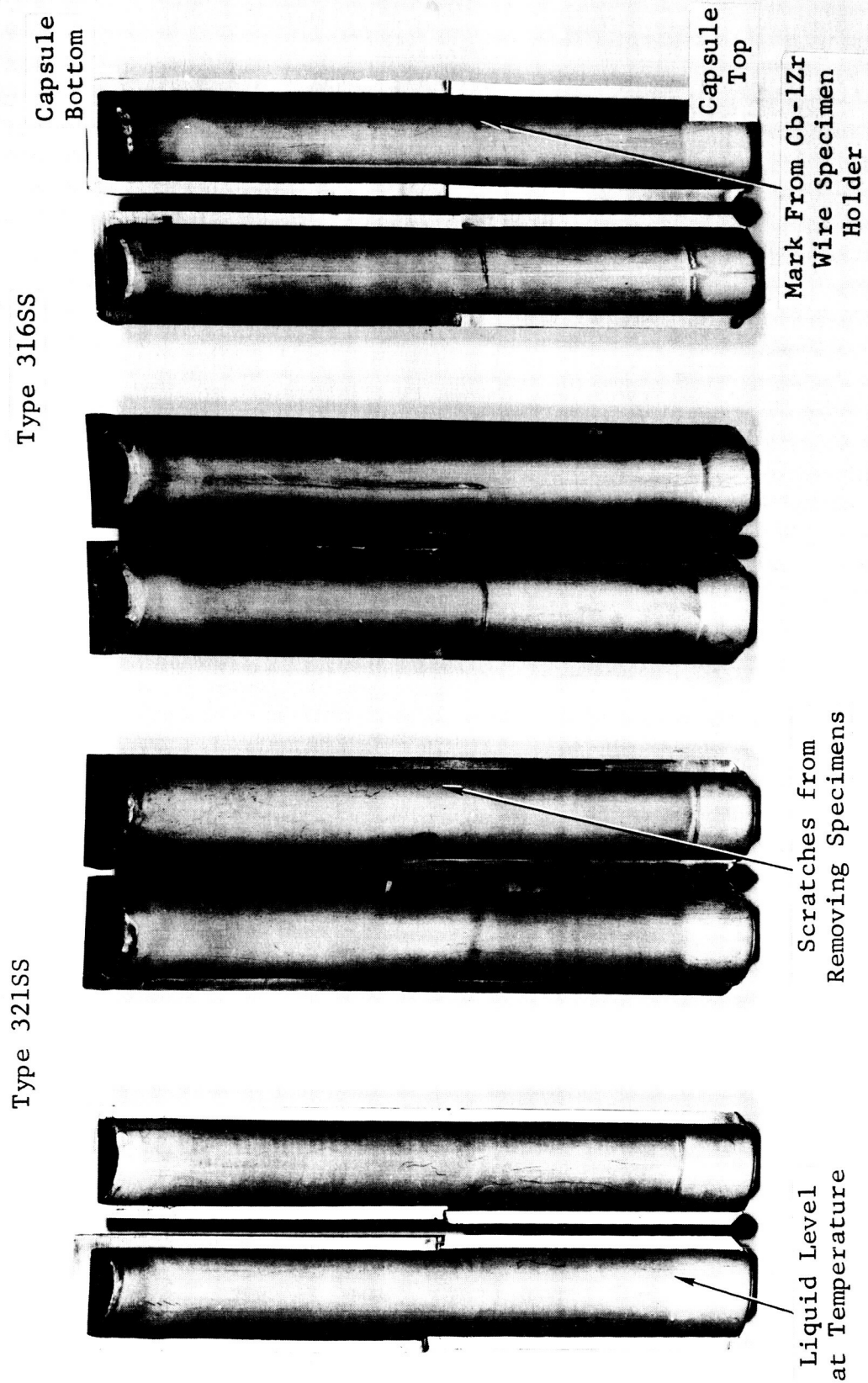


Figure 12. Sectioned Type 316SS and Type 321SS Isothermal Corrosion Capsules After Being Exposed to Potassium for 1,000 Hours at 1400°F.

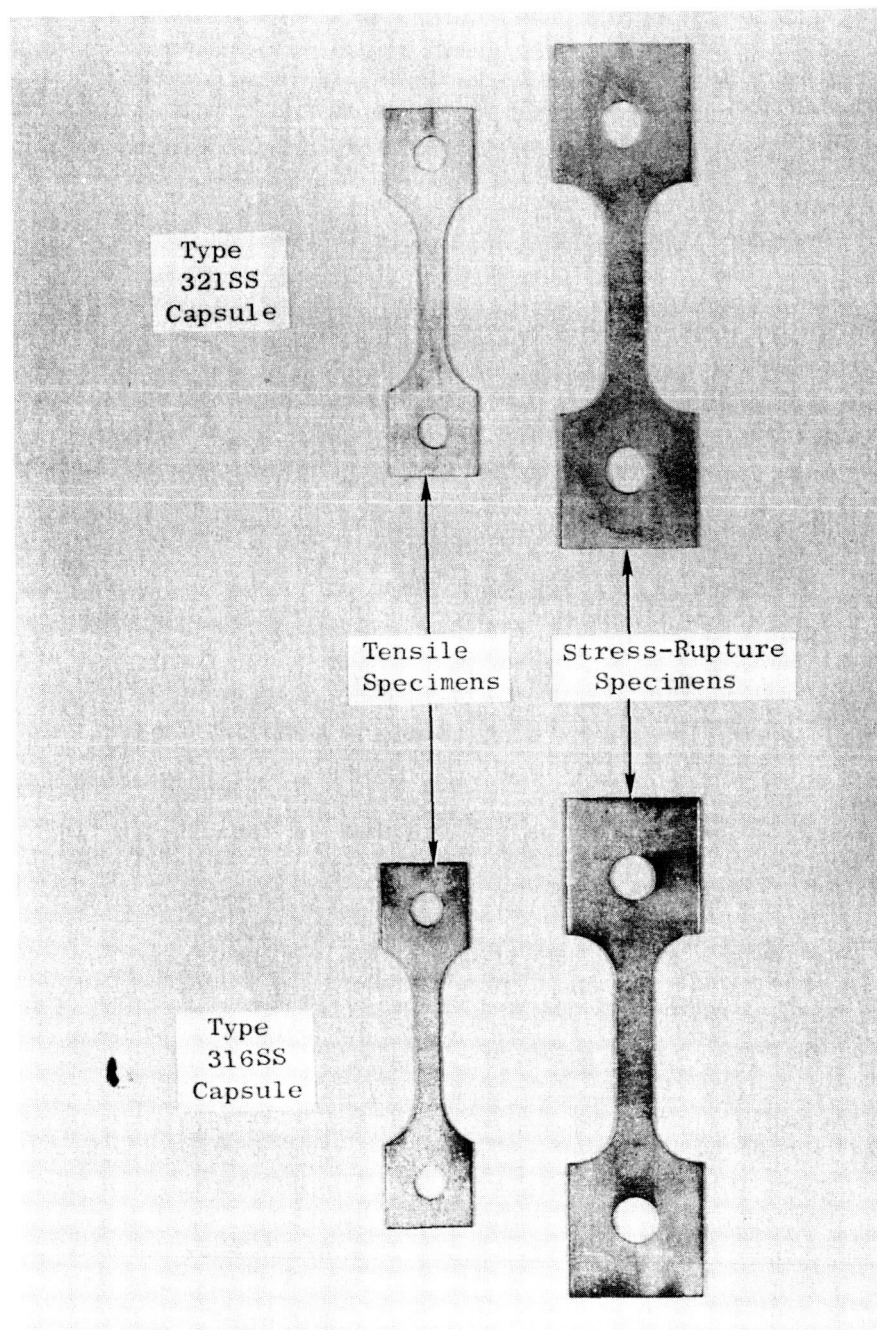


Figure 13.

Cb-1Zr Alloy Tensile and Stress-Rupture Sheet Specimens Exposed for 1,000 Hours at 1400°F in Potassium Contained in Type 316SS and Type 321SS Capsules. Definite Discoloration can be Noted on Specimens Exposed in a Type 316SS Capsule.

C65021112

TABLE I

POST-TEST CHEMICAL ANALYSES OF 0.040-INCH THICK Cb-1Zr ALLOY
SPECIMENS EXPOSED FOR 1,000 HOURS AT 1400°F IN POTASSIUM
CONTAINED IN TYPE 321SS AND TYPE 316SS CAPSULES

<u>Capsule Material and Specimen No.</u>	<u>Chemical Analyses of Cb-1Zr Alloy Specimens⁽¹⁾, ppm</u>			
	<u>C(2)</u>	<u>N(3)</u>	<u>O(3)</u>	<u>H(3)</u>
Type 316SS-1	210	206	258	23
Type 316SS-2	280	336	156	7
Type 321SS-3	50	21	147	13
Type 321SS-4	35	16	114	14
Pre-test-1 ⁽⁴⁾	10	14	75	6
Pre-test-2 ⁽⁵⁾	50	50	200	24

(1) MCN 454

(2) By Combustion Conductometric Techniques.

(3) By Vacuum Fusion Techniques.

(4) Conducted for Incoming Material Quality Control
Purposes on 9-8-64.

(5) Conducted as Companion Analysis at the Same Time
the Analyses were Performed on the Specimens Exposed
to Potassium (2-15-65).

contents were noted in the Cb-1Zr alloy specimens exposed in Type 321SS capsules as compared to the significant transfer of carbon and nitrogen that occurred in the Type 316SS capsules. There is no pronounced difference in the oxygen content of the Cb-1Zr alloy specimens exposed in either the Type 316SS or the Type 321SS capsules. The increased nitrogen and carbon contents of the Cb-1Zr alloy specimens tested in Type 316SS capsules significantly affected the room temperature tensile properties as evidenced by the higher yield strengths and the lower tensile elongation, Table II.

Expectations are that the results reported here have far reaching implications beyond the scope of this investigation. By appropriate employment of carbide-nitride forming alloying additions, such as titanium in Type 321SS, the transfer of nitrogen and carbon may be prevented or substantially reduced in a variety of systems and applications. A few examples of areas where this technique may be employed effectively are: 1) to reduce the outgassing of carbon and nitrogen from alloys exposed to high temperatures in high-vacuum systems, 2) to reduce the diffusion of carbon and nitrogen from the stainless steel to the refractory metal in coextruded tubing, and 3) to reduce the mass transfer of carbon and nitrogen that is induced by thermal gradients in certain monometallic systems which contain alkali metals. The latter case is of particular interest, inasmuch as carbon and nitrogen transfer have been observed in alloy steel, stainless steel, and superalloy containment systems. Presumably, the extent of this transfer could be reduced by alloying the containment material with stabilizing elements such as titanium.

TABLE II

ROOM TEMPERATURE TENSILE PROPERTIES OF 0.040-INCH THICK
Cb-1Zr ALLOY SPECIMENS⁽¹⁾ EXPOSED FOR 1,000 HOURS AT 1400°F
IN POTASSIUM CONTAINED IN TYPE 321SS AND TYPE 316SS CAPSULES

<u>Capsule Material and Specimen No.</u>	<u>0.2% Yield Strength⁽²⁾, psi</u>	<u>Ultimate Tensile Strength, psi</u>	<u>Elongation In/In, %</u>
Type 316SS-1	46,700	56,000	20
Type 316SS-2	40,300	57,000	15
Type 321SS-3	29,600	49,300	30
Type 321SS-4	25,300	42,800	36
Pre-test	23,300	40,000	37

(1) MCN 454

(2) Strain Rate 0.005 Inch Per Inch Through 0.2%
Yield Point.

V. FUTURE PLANS

A. Task I

1. Install tungsten caps on the Al_2O_3 probes and initiate the stress-corrosion capsule test.
2. Based on results obtained from this test, fill, seal, and initiate testing of a second capsule.

B. Task II

1. Complete the post-test evaluation for the bimetallic capsule program.

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